

N O T I C E

THIS DOCUMENT HAS BEEN REPRODUCED FROM
MICROFICHE. ALTHOUGH IT IS RECOGNIZED THAT
CERTAIN PORTIONS ARE ILLEGIBLE, IT IS BEING RELEASED
IN THE INTEREST OF MAKING AVAILABLE AS MUCH
INFORMATION AS POSSIBLE

9950-483

Unclas
41363

GENERAL ELECTRIC
ADVANCED ENERGY PROGRAMS DEPARTMENT
EVENDALE OPERATIONS
CINCINNATI, OHIO 45215

FINAL REPORT

A CONCEPTUAL DESIGN STUDY OF A
HIGH TEMPERATURE SOLAR THERMAL RECEIVER

(ADDED TASKS 6 AND 7)

BY

C.S. ROBERTSON, L.R. McCREIGHT, A. GATTI, AND H.W. SEMON

FOR

JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA 91103

M.L. PEELGREN
PROGRAM MANAGER

CONTRACT NO. 955455

NOVEMBER 7, 1980

GENERAL  ELECTRIC

ADVANCED ENERGY PROGRAMS DEPARTMENT
EVENDALE OPERATIONS
P.O. BOX 15132
CINCINNATI, OHIO 45215

"This work was performed for the Jet Propulsion Laboratory, California Institute of Technology sponsored by U.S. Department of Energy Through an agreement with the National Aeronautics and Space Administration."

"This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights."

ABSTRACT

Under JPL Contract 955455 a promising concept for a high temperature solar thermal receiver was studied. The key component of this concept is a coiled tube of silicon nitride which acts as a heat exchanger between the solar radiation and the pressurized working gas. The design appears to be ideal from the standpoint of utilizing structural ceramics at around 2500°F under severe thermal shock conditions. However the size and configuration of this coil are beyond the current state of the art for fabricating such materials as silicon nitride and carbide. A two-task program to develop and demonstrate the feasibility of extruding and forming a section of thin walled silicon nitride tubing was therefore undertaken as an addition to the original program. A promising polyvinyl butyral-based binder lubricant was identified. Fourteen full size extrusion experiments were conducted. Two trial firing of 1-1/4 turn helices were made. These trials were not entirely satisfactory; however, the lessons learned indicate that further work in the key processes may lead to a satisfactory fabrication procedure.

TABLE OF CONTENTS

	<u>Page No.</u>
ABSTRACT.	iii
SECTION I - SUMMARY	1-1
SECTION II - TECHNICAL RESULTS.	2-1
A. Fabrication Parameter Determination (Task 6)	2-1
1. Model System Analysis.	2-1
2. Full Diameter Extrusion Experiments.	2-1
B. Helix Fabrication (Task 7)	2-10
1. Fixtures and Jigs.	2-13
2. Final Extrusion Trials	2-19
3. Permeability Tests	2-25
4. Strength Tests	2-27
SECTION III - CONCLUSIONS AND RECOMMENDATIONS	3-1
SECTION IV - REFERENCES	4-1

LIST OF FIGURES

<u>Figure No.</u>		<u>Page No.</u>
2-1	Disassembled-Exploded View of Laboratory Extrusion Press Used During This Study.	2-2
2-2	Photographs of the Wahl Laboratory Extrusion Press Showing Control Console and Press in Operation .	2-4
2-3	Extruded Sections of 2 3/4" Diameter Tubing Using a Modified Wax Binder System	2-7
2-4	Burn-out Schedule for Removing Wax Binder-Lubricant System from GE128 Sialon Extruded Tubing . .	2-8
2-5	Thermo-Plastic-Thermal-Setting Binder-Lubricant System Developed by Coors Porcelain Company, Golden, Colorado.	2-9
2-6	A Sample of As-extruded With PVB 2.77 Inch O.D. Tubing, Prefired Tubing (to Remove Binder) and Fired Tubing.	2-12
2-7	Sketch of Helix Winding Mandrel	2-14
2-8	Photograph of the Helix Winding Mandrel	2-15
2-9	Mandrel Cutting Machine	2-16
2-10	Firing Fixture Concept.	2-18
2-11	1-1/4 Turn Helical Extrusion Positioned in Graphite Firing Fixture For First Trial Firing (July 18, 1980)	2-22
2-12	Firing Schedule for Trial	2-24
2-13	First Trial Firing Results.	2-26

LIST OF TABLES

<u>Table No.</u>		<u>Page No.</u>
2-1	Summary of Binder-Lubricant Formulations Used During Preliminary Studies.	2-3
2-2	Modified Binder - Lubricant System for 2 3/4" Tubing.	2-6
2-3	Modified Coors Thermo-Plastic Lubricant-Binder System Developed Here	2-11
2-4	Summary of Results of Firing 1-1/4 Turn Helix of GE 128 at GE Carbon Products to Approximately 1700°C in Nitrogen.	2-23

SECTION I

SUMMARY

Under JPL Contract 955455 a promising concept for a high temperature solar thermal receiver was studied.^{(1)*} The key component of this concept is a coiled tube of silicon nitride which acts as a heat exchanger between the solar radiation and the pressurized working gas. The design appears to be ideal from the standpoint of utilizing structural ceramics at around 2500°F under severe thermal shock conditions. However, the size and configuration of this coil are beyond the current state of the art for fabricating such materials as silicon nitride and carbide.

A two-task program to develop and demonstrate the feasibility of extruding and forming a section of thin-wall silicon nitride tubing was therefore undertaken. These tasks, numbered 6 and 7, represent additions to the original program.

In Task 6 several binder/lubricant systems were selected and tested for forming 1/2 inch diameter silicon nitride tubing in a small manually operated extrusion press. Although several of these compositions were successful, it did not prove to be as useful as anticipated to screen the compositions in this small size equipment. This was due to scale up and operating parameters that were more easily determined in a hydraulic press capable of extruding full size tubing.

Of the binder lubricant compositions tested, the aqueous base varieties were found to be unsuitable without extensive further development. However, two organic thermoplastic types were quite successfully used to make short articles for demonstration of feasibility. These were a mixed wax composition and a polymeric rubber, polyvinyl butyral. The wax composition was quite difficult to remove from the formed tubing without slumping. It could be removed in a vacuum furnace only over an impractically long time period. The PVB on the other hand can be removed

*References are listed in Section IV.

relatively easily in an air atmosphere furnace leaving a handleable, fragile shape to be further fired separately in the laboratory nitrogen atmosphere furnace.

PVB type binders should preferably be mixed hot (150-160°C) in a high shear type of blender or mixer in order to uniformly distribute them over the particles. They also should be extruded hot. In the absence of suitable equipment for performing these two operations hot, modifications to the usual procedures were made by using isopropanol to dissolve and lower the viscosity of the binders. This was a partially successful procedure which permitted forming some short lengths of quite good tubing.

Task 7 was then directed towards scaling up the work from Task 6 to enable fabrication of sections of full-size helices. These sections would consist of about 2-1/2 turns of 2.3 inch diameter 0.10 inch wall tubing. The overall helix diameter would be about 10 inches center-to-center.

A winding jig onto which the extruded tubing could be wound and supported for drying was fabricated from plywood and polystyrene foam. It is large enough to hold 2-3 experimental extrusions or one eventual full size extrusion.

Numerous (14) extrusion experiments were conducted, using primarily the PVB binder lubricant system. These were intended to extrude the full size tubing and form it into a helix of up to 2-1/2 turn pieces. The work yielded several pieces of approximately half the desired length but not without some deformation and other defects. The insufficiently homogeneous mixture of PVB and silicon nitride was part of the problem. However, the working time of these compositions was also very short (at least partially due to the volatility of the alcohol). Tubing would therefore tend to be either too soft or too hard with the result that it would easily deform or not be formable into a helix without cracks.

Many good short lengths of both straight and curved tubing were made and fired in the laboratory scale nitrogen furnace. As an adjunct

to this work, some tubes were also glazed with a refractory glass that may provide a desirable benefit (e.g., corrosion or oxidation resistance) in a future application. Leak testing of the glazed tubes was performed.

A graphite firing jig was designed and built to hold a 2-1/2 turn coil with provision to accommodate the expected 16-18% shrinkage. Both the winding and the firing jigs were constructed in a manner that permitted disassembly from the inside of the helical coil.

The firing of these large coils was originally planned to be performed in our sister division's Northeast Philadelphia "D St." Laboratories. The furnace which we planned to use was damaged in an accident unrelated to this program. The power supply available after the accident was not capable of heating the coil rapidly enough to produce acceptable results or to avoid contamination of the furnace. Alternate furnaces were then located at General Electric's Carbon Products Department at East Stroudsburg, PA and used for two firings. These furnaces are large induction heated units normally used for firing large loads of carbon blocks to much higher temperatures than required for the silicon nitride coils of this program. The firings were therefore quite difficult to control, and the results were not entirely satisfactory; however, with further experience and modifications in the equipment and procedures we would expect to be successful.

An additional problem with the firing procedure was that, to minimize handling the large sections of coil too much, it was decided to burn the binders out in the same furnace as the final firing during the initial part of the cycle. This proved to be difficult to control in these particular furnaces.

SECTION II

TECHNICAL RESULTS

A. FABRICATION PARAMETER DETERMINATION (TASK 6)

1. Model System Analysis

Preliminary experiments were made using a laboratory extrusion press shown in Figure 2-1. This press was assembled with a die and mandrel combination which formed a tube 1/2 inch OD with a 1/16 inch wall thickness. Table 2-1 summarizes the results of this phase of the study which served to both model the full size equipment which had been ordered and eliminate obviously unsuitable binder-lubricant systems. The aqueous systems shown on Table 2-1 were not viable systems nor were schemes to shield the powders from aqueous systems applicable without further development. It was concluded that only nonaqueous approaches should be pursued and those systems were studied using a full scale die. The model system also showed that the extrusion parameters such as extrusion ratio, ratio of coil diameter to tube diameter, and ratio of tube diameter to tube wall thickness favored the forming of the larger diameter tubes rather than small tubes.

Experimental results showed that this conclusion was correct in as much as large diameter tubing was formed with slight process modifications without undue difficulties. Other factors such as fabrication of the green state helix, unfired tubing deforming under its own weight, and firing process conditions were found to be the technical factors requiring control.

2. Full Diameter Extrusion Experiments

Two and three-quarter inch diameter tubing with 0.125 inch thick walls was extruded with the Wahl press shown in Figure 2-2. The binder-

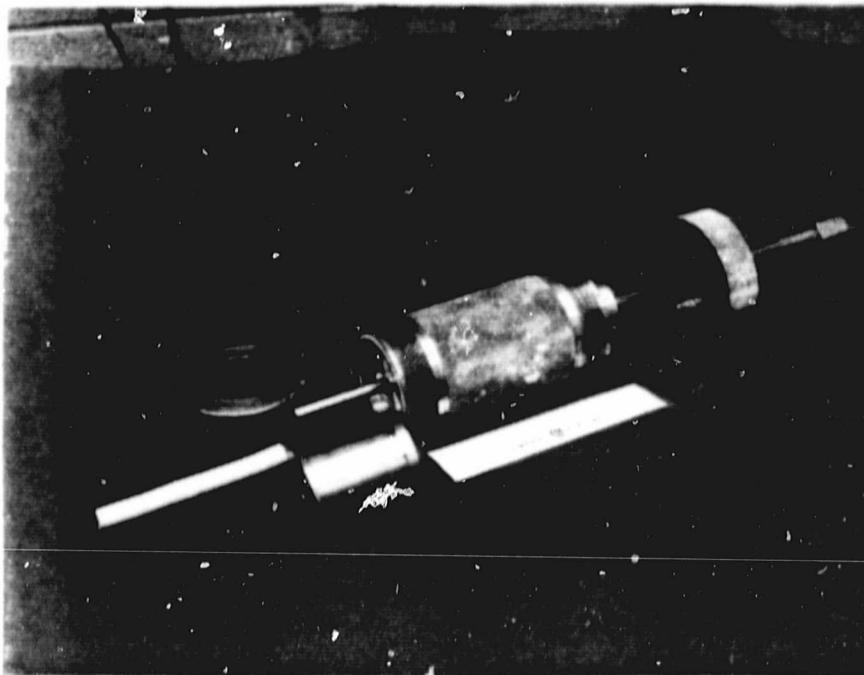


Figure 2-1. Disassembled-Exploded View of Laboratory Extrusion Press Used During This Study.

ORIGINAL PAGE IS
OF POOR QUALITY

TABLE 2-1

SUMMARY OF BINDER-LUBRICANT FORMULATIONS USED DURING PRELIMINARY STUDIES

Recipe #	Composition												Remarks
	H ₂ O	Oleic(a) Acid	Butyl Sterate	Reten	PVA	S	MFO	Isopropyl Alcohol	PVB	P. Glyc.	DOP	Glyc.	
617	800 cc	yes	76 cc	16 gm									Dilalent
619	800 cc	no	76 cc	16 gm								100 cc	Dilalent
620								100 cc					No green strength
623							9.5 gm	500 cc	22 gm	24 gm	20 gm		Not plastic enough, dries too quickly
627	200 cc	yes			20 gm								Dilalent
628								500 cc	50 gm				Not plastic enough
635							9.5 gm	500 cc	22 gm	24 gm	20 gm	55 cc	Thixotropic, good green strength. Extrudes well

(a) Oleic acid added by dry milling 500 gms of GE-128 sialon with 5 gms oleic acid.

PVA - Polyvinyl alcohol

S - Orange shellac

MFO - Menhaden fish oil

PVB - Polyvinyl Butyral

P.Glyc. - Polyethylene glycol

DOP - Dioctyl Phthalate

Reten - Polyacoylamide

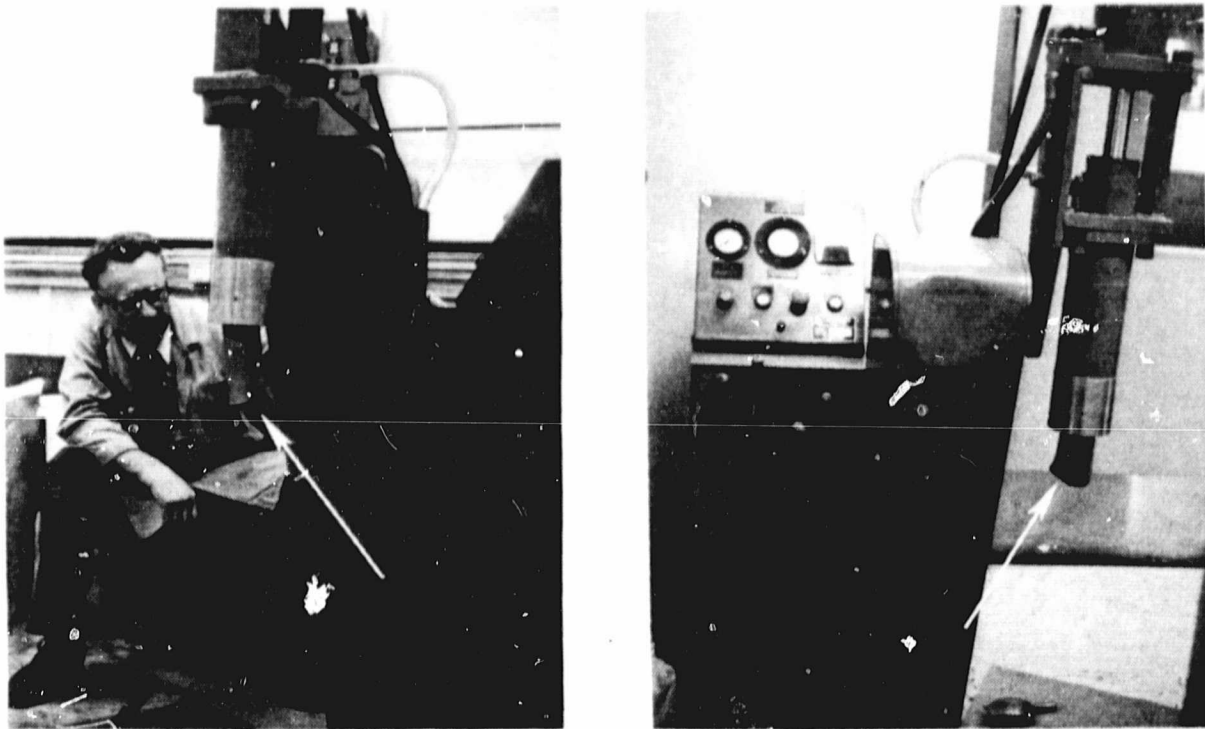


Figure 2-2. Photographs of the Wahl Laboratory Extrusion Press Showing control Console and Press In Operation.

ORIGINAL PAGE IS
OF POOR QUALITY

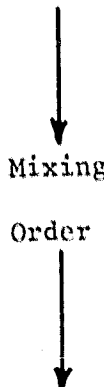
lubricant mix (#635 from Table 2-1) previously found to be usable for 1/2 inch tubing proved to be too fluid for the larger diameter, lower extrusion ratio tubing. The mix was further modified with glycerine as shown in Table 2-2. This material extruded with limited success. Green tubing could be extruded, dried, and fired successfully. However, handling the green ware prior to drying was difficult because of its highly thixotropic nature and tendency to change shape easily if moved. Efforts to decrease thixotropy were not successful. Stiffer mixtures would not extrude due to the maximum pressure limits of the press. (2500 psi).

a. Wax-Based Binder Lubricant System. The system using mixed waxes developed for another program was modified by reducing the wax content markedly. The 1/2 inch diameter laboratory extruder would not extrude mixtures with over 40 volume percent solids. However, the 2-3/4 inch diameter system will easily extrude 55% by volume solids mixtures. These higher solids mixtures can be more stable during binder removal and thus are less likely to slump or deform. Figure 2-3 is a photograph of pieces of 2-3/4 inch tubing extruded using these wax mixtures. The half circle section of 16 inches in diameter was fabricated by adjusting the mandrel position in the die body with built-in positioning screws. As can be seen, excellent straight and curved sections can be extruded with this mixture.

The firing schedule shown in Figure 2-4 removed the binder without slumping the tubing. As can be seen, the procedure is tedious, and did not appear to be a practical production process. Further work on the wax system was not performed because an apparently more workable binder-lubricant system was selected and developed.

b. Thermoplastic Binder-Lubricant System. A thermoplastic binder-lubricant system developed by Coors⁽²⁾ was studied. The advantage of this approach is that parts of the binder-lubricant system can be removed by solvent extraction while simultaneously converting some of the binder to a thermo-setting plastic. This prevents slumping during subsequent firing. Figure 2-5 is a flow diagram of the process.

TABLE 2-2MODIFIED BINDER - LUBRICANT SYSTEM FOR 2 3/4" TUBING

 Mixing Order	1. Isopropyl Alcohol	250 cc
	2. Polyvinyl Butyral (PVB)	22.2 gms
	3. Glycerine	250 cc
	4. Menhaden Fish Oil	9.44 gms
	5. Polyvinyl Glycol	23.9 gms
	6. Dioctyl Phthalate (DOP)	19.9 gms

900 cc to 2500 gm GE-128 (825 cc) 48 V/O solids

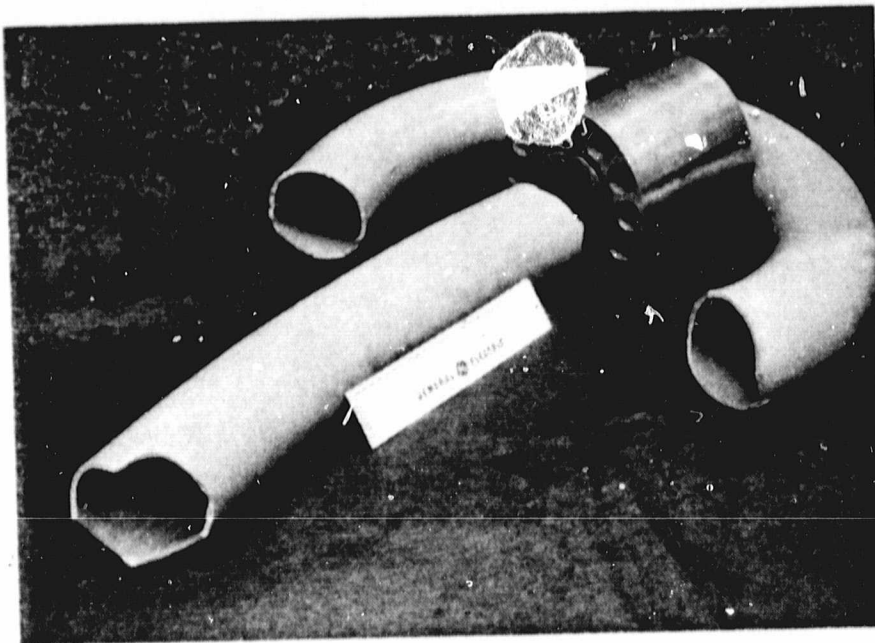


Figure 2-3. Extruded sections of 2-3/4" diameter tubing using a modified wax binder system

ORIGINAL PAGE IS
OF POOR QUALITY

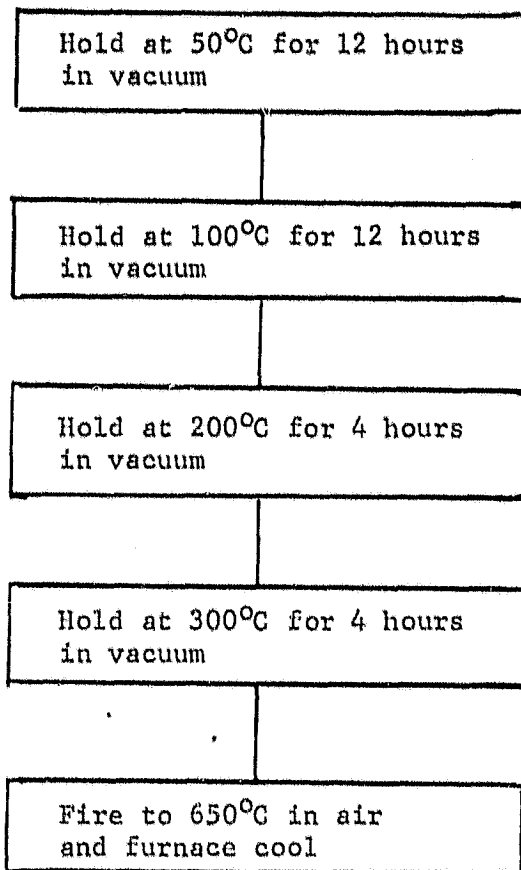


Figure 2-4. Burn-out schedule for removing wax binder-lubricant system from GE128 sialon extruded tubing.

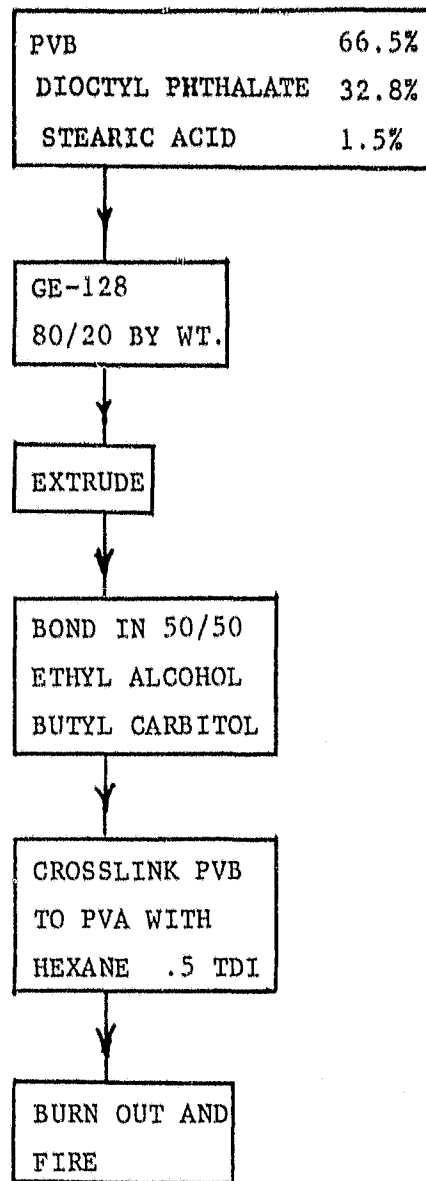
THERMO-PLASTIC SYSTEMS

Figure 2-5. Thermo-plastic-thermal-setting binder-lubricant system developed by Coors Porcelain Company, Golden, Colorado.

Unfortunately, this thermo-plastic-polymerizable system proved to be too stiff at room temperature for the available extrusion equipment. A 5/16 inch diameter bolt which holds the mandrel-spider assembly repeatedly failed after short pieces (2 in. long) were extruded. The extrusion pressure required was in excess of 2000 lbs. which translates to 72,400 psi on the bolt (well above its tensile strength).

Since this extrusion binder system could not be directly used with the equipment on hand, it was decided to adjust its plasticity with isopropyl alcohol rather than elevated temperature. The approach appeared to be quite successful and satisfactory short extrusions were made routinely. Table 2-3 shows the formulation developed.

This binder-lubricant mixture produced 2-3/4 inch diameter sialon tubing with a 0.125 inch wall thickness which did not easily deform during extrusion and generally dried without cracking. The binder was removed readily by air firing at 50°C/hr to 300°C and then to 650°C in one hour. The tubing was then final fired in nitrogen at 1750°C for 30 minutes.

Figure 2-6 shows samples of as-extruded and fired pieces of GE128 tubing.

All of the subsequent effort to produce an acceptable helical coil of 2 to 3 turns from extruded GE128 sialon was made using the modified Coors extrusion binder formulation. However, as will be described below, the press capacity and lack of extrusion flow control together prevented the achievement of the goal of fabricating the desired demonstration hardware.

B. HELIX FABRICATION (TASK 7)

The alcohol modified PVB binder-lubricant system was chosen for the experiments aimed at scaling up the extrusion tests to produce 2-1/2 turn sections of helix. Suitable jigs for winding and firing these parts were needed and the design of them is described next.

TABLE 2-3MODIFIED COORS THERMO-PLASTIC LUBRICANT-BINDER
SYSTEM DEVELOPED HERE

Components	Quantity	
	CC	Gms.
GE128 Sialon*		2300
Isopropyl Alcohol	445	
Polyvinyl butyral		197
Dioctyl Phthalate	97	
Stearic acid	6.5	

*Solids content by volume 52%

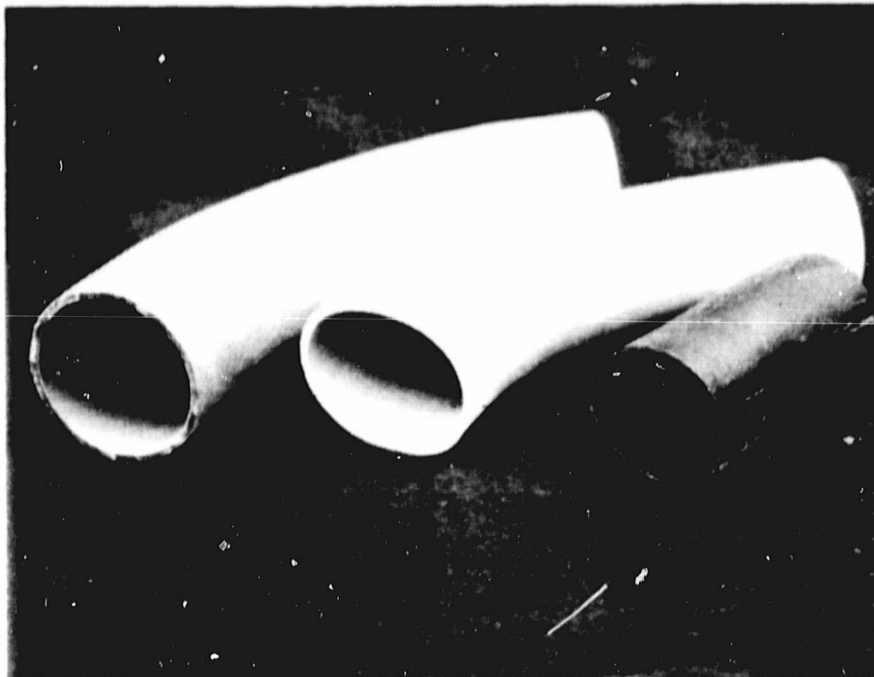


Figure 2-6. A sample of as-extruded with PVB 2.77 Inch O.D. tubing, prefired tubing (to remove binder) and fired tubing are shown, left to right.

ORIGINAL PAGE 2
OF 1000

1. Fixtures and Jigs

Some of the technical challenges in the development of a helical heat exchanger are in the design of suitable fixtures for initially forming the coil and for holding it in the proper shape during firing. One fixture must accept the ceramic tube as it comes from the extrusion press, hold it during winding, and finally, support it while the solvent dries out and the coil becomes self supporting. This is more difficult than it sounds, because the green ceramic shrinks as it dries and has little tensile strength in the green process state. The design of the fixture which holds the coil during firing is further complicated in that the fixture must be able to accommodate a shrinkage of 16% and must withstand a firing temperature of 1750°C.

a. Winding Mandrel. We determined that the requirements for the winding mandrel could best be met by making it of wood and Styrofoam®. Styrofoam® is inexpensive, is easy to machine, is strong enough to withstand winding forces, and is resilient enough to accommodate the shrinkage that occurs during solvent evaporation. A Styrofoam® and wood mandrel is shown schematically in Figure 2-7 and photographically in Figure 2-8. It is designed so that the actual winding is on the Styrofoam® mandrel but the wood provides the bulk of the structural strength. Note that the mandrel is supported on three rollers that are positioned so as to not only support the mandrel but also to support the underside of each turn of the coil.

Building the mandrel presented a challenge because lathes or other machine tools that can handle a 22 inch diameter cylinder approximately 39 inches long, and machine a 2-3/4 wide 2-3/4 deep helical groove with a 3-3/4 inch pitch, thereon, are items of limited availability. This was solved by designing a special machine, made primarily of wood, several pieces of "Unistrut", and a high speed vacuum cleaner motor. This device successfully produced the Styrofoam® mandrel. This machine is shown schematically in Figure 2-9. It must be emphasized that building and using this machine would have been substantially more difficult if it had been necessary to use a material more difficult to machine

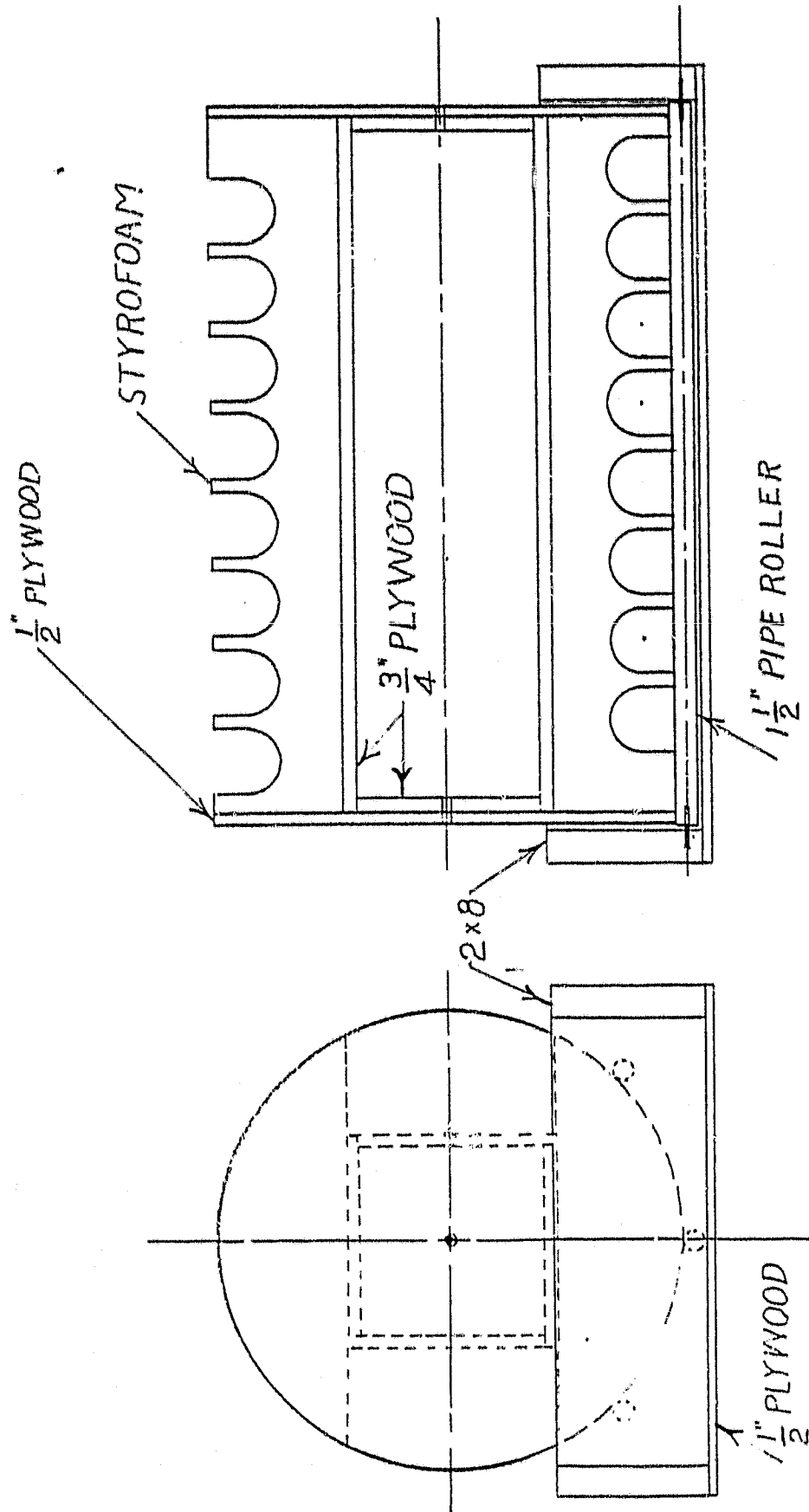


Figure 2-7. Sketch of helix winding mandrel.

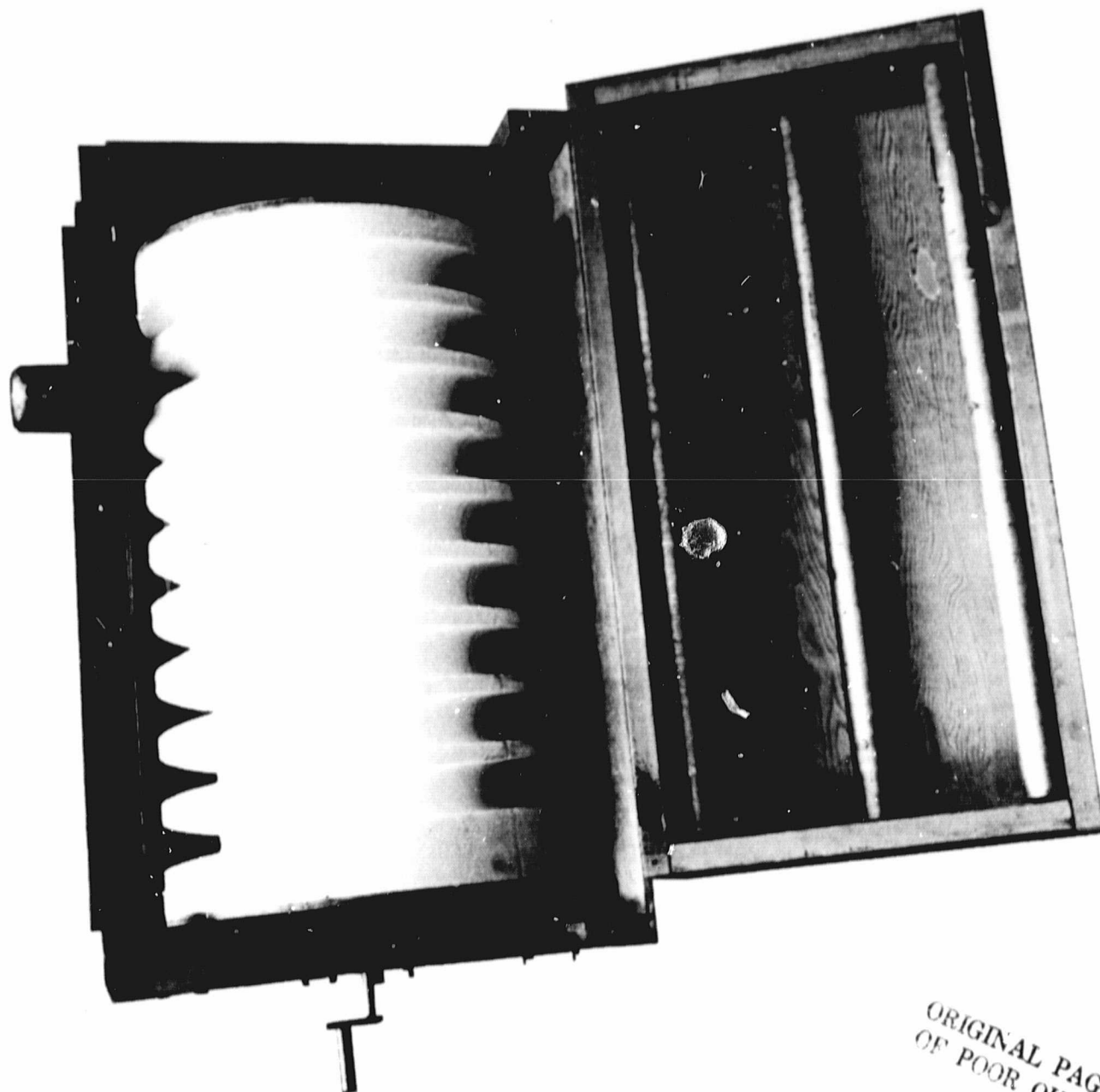


Figure 2-8. Photograph of the Helix Winding Mandrel

ORIGINAL PAGE IS
OF POOR QUALITY

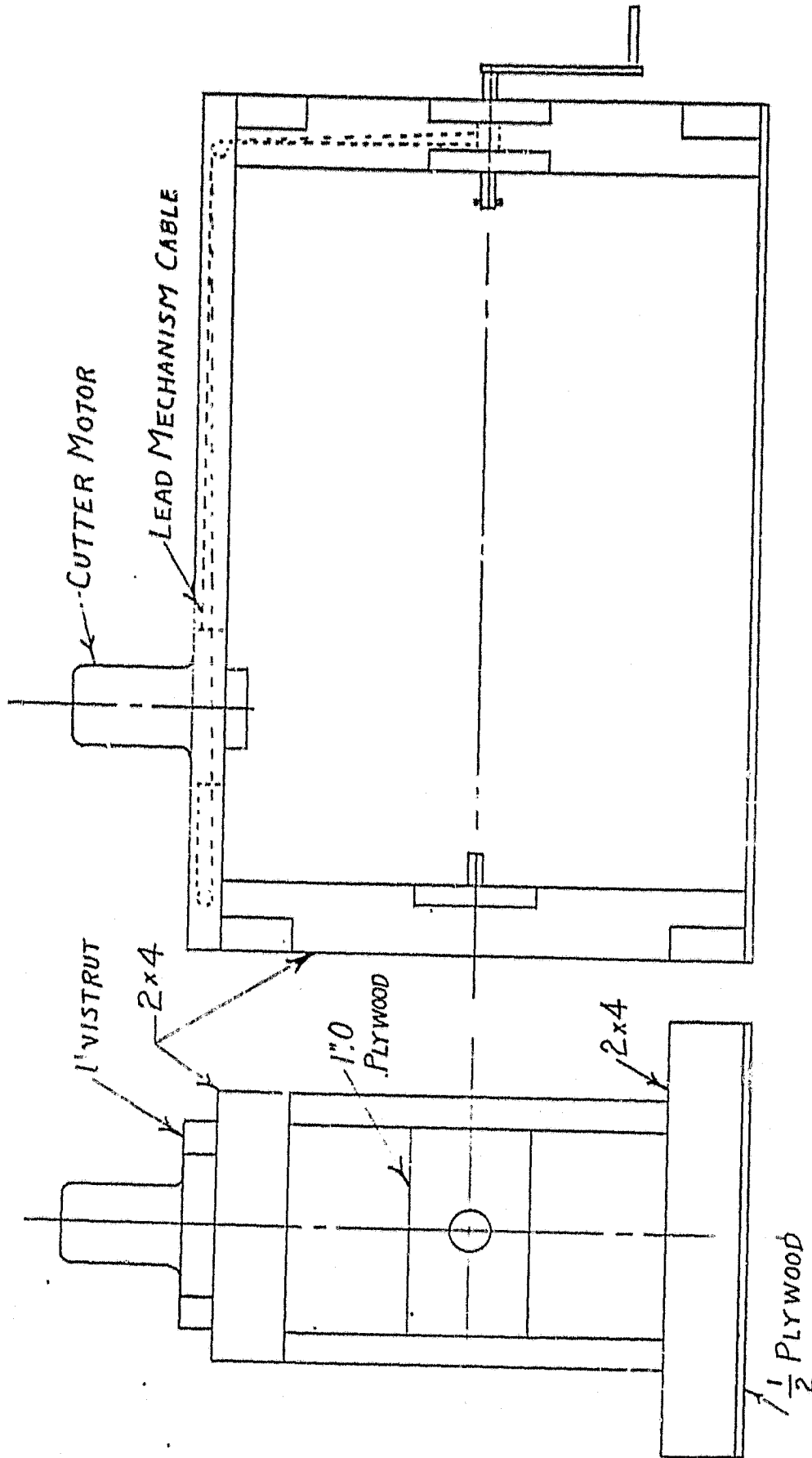


Figure 2-9. Mandrel Cutting Machine

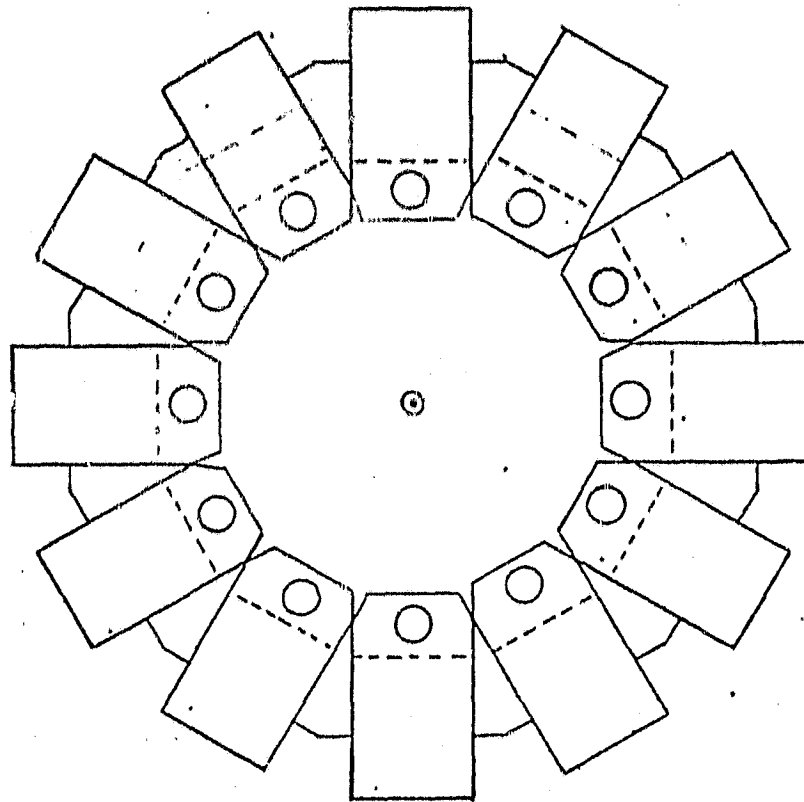
than Styrofoam®. The coil winding fixture was used at the GE Switch Gear site to produce coiled parts.

b. Firing Fixture. In the design of the firing fixture there are few material choices due to the firing temperature. Graphite was the selected material which precludes the use of a simple wooden machine to cut the necessary helical groove. Therefore, a semi-skeleton support fixture was designed that consists of 12 comb-shaped pieces, supported by disks at either end, in which the helical groove is approximated by a series of properly spaced and angled cuts in the comb pieces. This makes it possible to do the bulk of the machining on a band saw at considerably lower cost than would be required to form the helix by continuous machining.

The comb type firing fixture is shown schematically in Figure 2-10. Shrinkage is accommodated in this fixture by making the cutouts in the combs deep enough so that the coils simply move into the cutouts as shrinkage occurs with no restraint other than friction.

This comb type firing fixture is held together by an axial bolt and plate arrangement, made of steel, which can be removed after the fixture is installed in the furnace but before firing. This bolt is designed to be reinstalled after firing, to facilitate removal from the furnace and for subsequent handling.

Using the comb type fixture, which goes into the furnace with its axis vertical, makes it possible to utilize a furnace with a working diameter only slightly in excess of 22 inches. Consideration was given to the design of a fixture arrangement in which each coil in the helix would be supported over much of its bottom half by a suitably shaped graphite boat. This system would require that the coil be fired with its axis horizontal. High temperature, controlled atmosphere furnaces tend to be cylinders, with their axes vertical and with length to diameter ratios in the order of 1-1/2 or 2 to 1. Thus it would take a very much larger furnace to fire the coil with its axis horizontal. For



Top View - Top Plate Removed

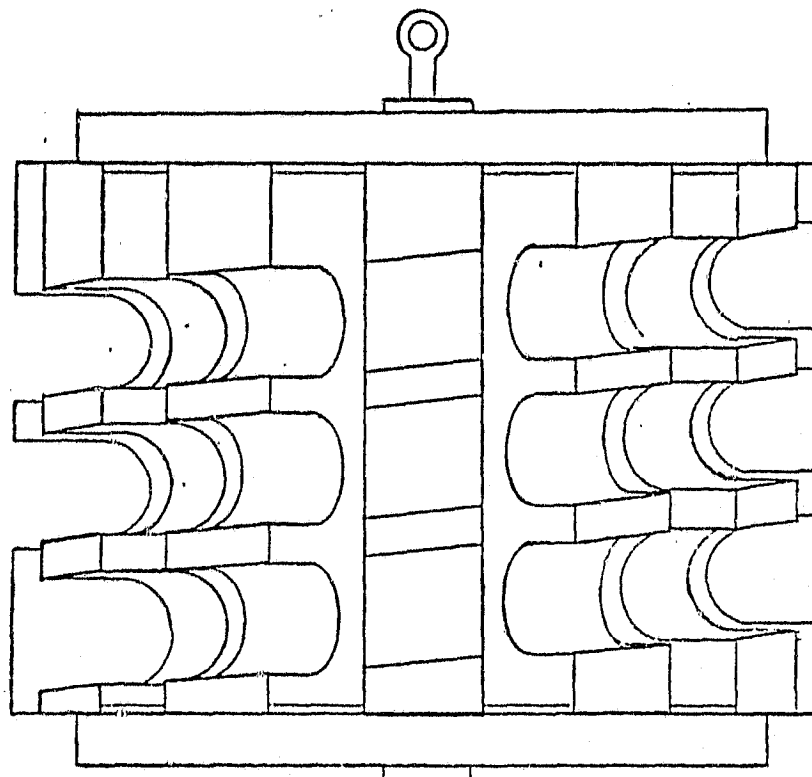


Figure 2-10. Firing Fixture Concept

this reason, and because the boat type fixture was judged to provide inferior support and to be more expensive to make, it was decided to use the comb style fixture. A photograph of the actual fixture fabricated is shown later in this report as Figure 2-11.

2. Final Extrusion Trials

A total of fourteen runs were made during the final month of this study. During this time it was found that:

1. The original equipment required strengthening to accommodate higher extrusion pressures.
2. The extrusion press hopper capacity was inadequate and it could not be reloaded quickly enough to make long lengths of defect free tubing when multiple consecutive batches of feed stock were used. Sufficient length tubing for only about 1-1/4 turns of the helix can be extruded using present operating parameters.
3. Extrusion mixes as prepared in the muller type mixer were not completely homogenous. It was found that the mix can be made more plastic and homogeneous if allowed to age at least 16 hours in a double plastic bag before using. This aging appears to achieve equilibrium mixing of the alcohol by diffusion and, possibly, crosslinking of the PVB.

At least four of our total of fourteen runs were aborted due to mechanical failure of the spider system which holds the extrusion mandrel in place. The final solution was to use a 7/16 inch-14 allen head cap screw to bolt the mandrel to the spider and to have the spider itself rewelded using stainless steel welds with good penetration. Once this was done, extrusion pressures to the limit of the press capacity (2500 psi) could be used without mechanical failure.

Mixing and extruding material on a same-day schedule allowed adjustments to be made in the viscosity of the mix but limited the homo-

geneity and plasticity. However, it also limited our ability to try more modifications because of the need to burn-out, mill, and screen our reusable material. It was decided to modify the liquid content of the mix by qualitative judgement once the basic amounts of isopropyl alcohol, PVB, DOP, and stearic acid were milled into the GE128 powder and age the resulting mixes at least 16 hours before use.

Four distinct appearances of a mulled mix could be reproduced reliably and their extrusion behaviors correlated.

1. "Sandy" beads of material between 1/4 in. diameter to 1/2 in. diameter. Extrusion mix in this condition extruded at pressures between 2000 and 2200 pounds and made dense, hard excellent tubing which held its shape without slumping (could be laid on a flat). This material had no excess plasticity and could not be shaped around the mandrel without cracking.
2. "Marbles" balls of material 1/2 in. diameter to 1-1/2 in. diameter. Extrusion mix in this condition extruded at pressures between 1500 and 1600 pounds and made moderately hard, dense excellent tubing which held its shape with minimal slumping (could be laid in a trough), but had little excess plasticity and cracked if bending was attempted.
3. "Lumpy" balls 1-1/2 in. diameter to 6 in. diameter. Extrusion mix in this condition extruded at pressures between 1000 pounds and 1100 pounds and made soft, dense excellent tubing which slumped slightly when formed in a trough. Material in this condition could be bent to the radius of curvature of our forming mandrel without cracking.
4. "Stringy" elongated sausage shapes which finally end up as one large lump. This material extrudes at pressures between 500 and 700 pounds, is very soft, and slumps to the shape of a trough with a flat top. This material can be easily bent without cracking but distorted badly in the mandrel winder

and usually failed in tension hanging by its own weight on the underside (gravity side) of the winder.

The ability to extrude more than about 1-1/4 turns was hampered by the necessity of recharging the press. During the time-interval it took to recharge, enough drying of the original extrusion occurred to form a defect at the die opening which cracked while being wound. The combination of press and mix was thus limited to about 1-1/4 turns rather than the 2-1/2 turn goal.

a. Extrusion Results. 1-1/4 turn extrusions were made using mix No. 3 ("lumpy") although mix #2 ("marbles") would be more desirable because of its shape retention ability. However, flow control of the extrusion process was poor so that coil shapes could not be formed directly as we had hoped. Mix #3 was pliable enough to shape into the helix with the winding mandrel in a vertical position thus minimizing gravity effects on the green formed material.

b. Firing. A test firing at our Reentry Systems Division D Street Facility was done in a small furnace using a similar schedule to the one planned to be used in the large furnace for full diameter helical coils. The large furnace had recently been accidentally damaged and is being operated temporarily with a much smaller-than-optimum power supply which markedly lengthens the firing schedule.

The results of this test firing indicated excessive shrinkage and evaporation of the silicon nitride which in turn contaminated the furnace lining. This contamination caused the owner to forbid us to use his large furnace unless we could install a separate liner for our work. This solution would have caused schedule delays for both the furnace operator and for us. Even more important to us was the serious evaporation of silicon nitride caused by the long firing schedule. For these reasons an alternate furnace was sought. A satisfactory one was located at the GE Carbon Products Department in East Stroudsburg, PA. Two experimental firings were done there. Figure 2-11 shows the graphite firing fixture holding about a 1-1/4 turn helical coil of GE128 sialon. This assembly was transported to GE Carbon Products and fired at their facility. Table 2-4 and Figure 2-12 summarize the firing schedule used.

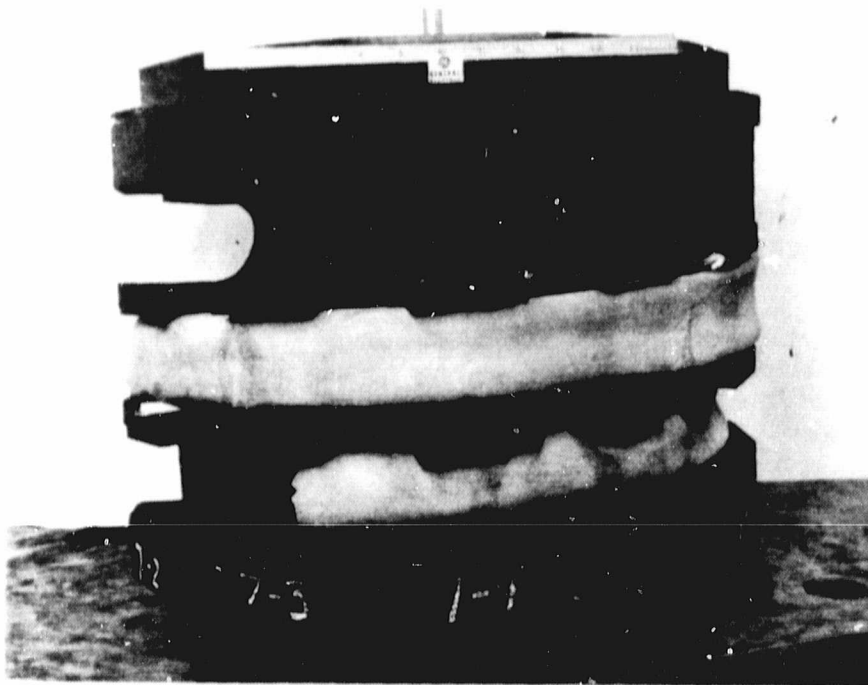


Figure 2-11. 1-1/4 Turn Helical Extrusion Positioned in Graphite Firing Fixture for First Trial Firing (July 18, 1980)

ORIGINAL PAGE IS
OF POOR QUALITY

TABLE 2-4

SUMMARY OF RESULTS OF FIRING 1-1/4 TURN HELIX OF GE 128
AT GE CARBON PRODUCTS TO APPROXIMATELY 1777°C IN NITROGEN

Time Minutes	Power		Power Level (200 KW Max.)	Remarks
	On	Off		
0	X		50%	
5		X		Began Smoking
35	X		50%	Smoking
39		X		
49	X		50%	
54		X		
65	X		50%	
69		X		
74	X		50%	Less Smoke Now
82	X		50%	
89	X		50%	Smoking Has Stopped
89	X		50%	Red
91	X		70%	
93	X		70%	1050°C
95	X			1100°C
99	X			1150°C
102	X			1225°C
106	X			1285°C
109	X			1350°C
110	X			1400°C
118	X			1475°C
122	X		70%	1600°C
127	X		40%	1700°C
132	X		35%	1750°C
124	X		35%	
137	X		35%	1650°C
138	X		50%	
139	X		50%	1700°C
142	X		50%	

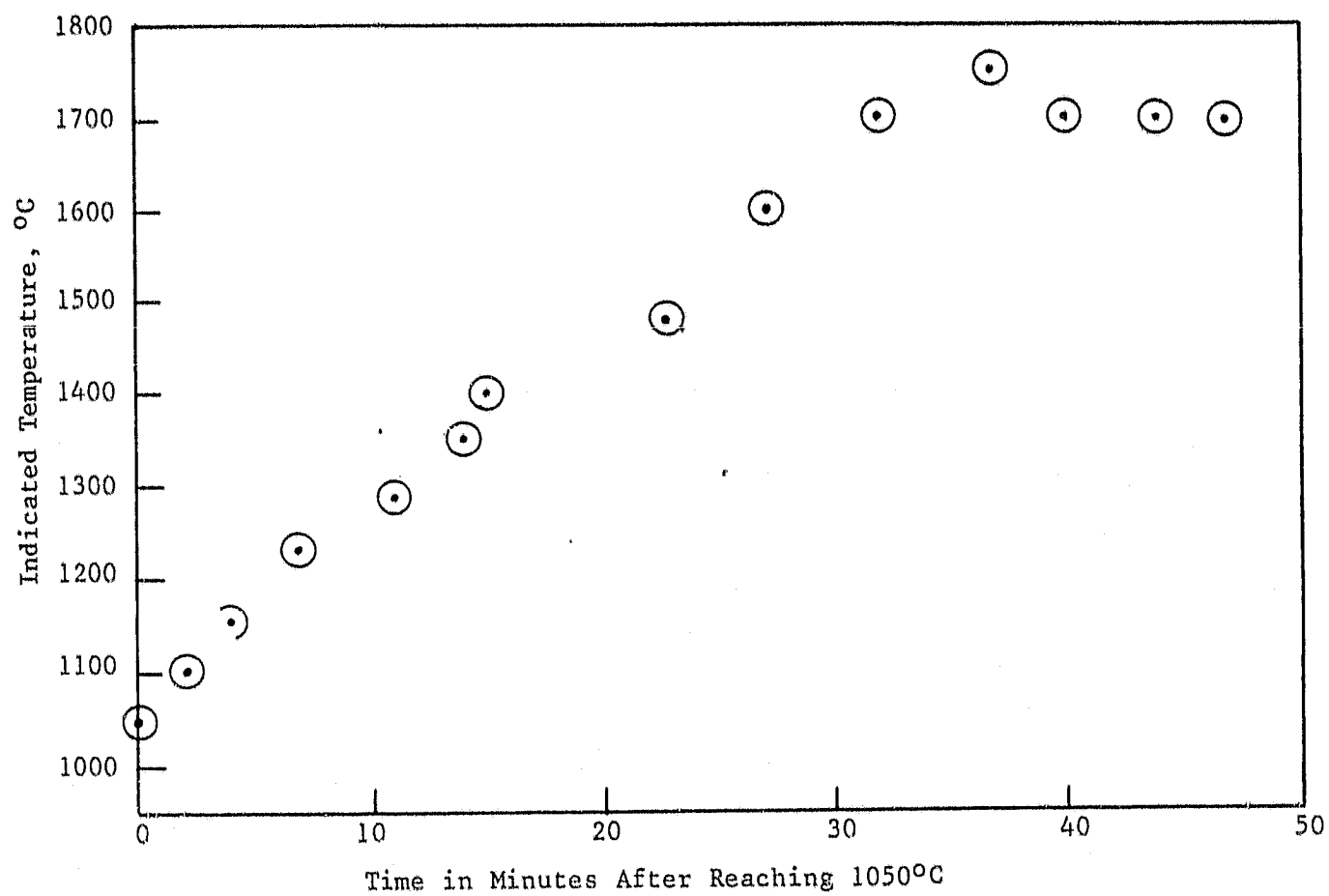


Figure 2-12. Firing Schedule for Trial

Power was jogged during the initial warm-up to facilitate binder burnout. Once smoking stopped, the power was increased to 70% of capacity until sintering temperature was reached. Binder burnout was effected in one hour and forty minutes, and sintering temperature (1700°C) was reached forty minutes later. The sintering temperature was maintained for only 20 minutes rather than the usual 30 minutes because heavy volatilization of the material was observed. The furnace was then shut down and allowed to cool at its normal rate. Temperature measurement was accomplished using a Leeds and Northrup optical pyrometer sighting on a mirror over a vent hole in the furnace. This mirror continually fogged and thus distorted the readings. Upon cooling, the fired helix was examined and found to have vaporized considerably, collapsed somewhat, and cracked in three places. It was apparent from these results that the material had considerably overfired. Figure 2-13 shows the results of this firing.

Our second and final firing attempt was made using a nitrogen cooled direct viewing port attached to the furnace crucible rather than using the open port and mirror system described earlier. This modification proved very satisfactory in that much more reliable temperature monitoring was possible. Still, the excess power available due to the large scale and high temperature design was a factor in also slightly overfiring this helix. It was apparent from this second firing that more control would be needed to fire components to optimum temperatures. Nevertheless, the coil from the second firing was much improved in appearance and integrity. The 1-1/4 turn helix had fewer cracks and was less damaged by overvaporization than the first trial test part.

It is believed, based on these latest results, that the firing technique can be controlled to produce acceptable helices.

3. Permeability Tests

Pressure testing of 8 inch long sections of fired tubing showed that the structures were not gas tight due to local defects which are aligned with the extrusion direction of the tubing. These defects presumably arise from incomplete homogenization of the extrusion mix which allowed binder-rich areas to occur. During burn-out these defects be-



Figure 2-13. First Trial Firing Results

ORIGINAL PAGE IS
OF POOR QUALITY

come areas of excess porosity that leak under pressure. Mixing techniques were varied to minimize these porous areas, but were not entirely successful using a single-wheel, muller-type mixer.

Glazing of the sintered silicon nitride tubes using a very refractory glass, which is compatible with the silicon nitride from a thermal expansion standpoint has produced impermeable tubes as determined by water bubble tests. This glazing operation may have other desirable benefits such as providing oxidation protection.

4. Strength Tests

As another adjunct to this program, the strength of GE128 sintered silicon nitride was briefly rechecked at room temperature and at 1400°C. These results compared favorably with other work reported on silicon nitride.

Four-point load tests of the GE128 silicon nitride show an average strength of 42,000 psi (39-46K) at room temperature and 22,000 psi at 1400°C with only a 5% variation in strength.

SECTION III

CONCLUSIONS AND RECOMMENDATIONS

The helical design has been identified as an excellent configuration for construction of the heat exchanger for a high temperature solar receiver built of structural ceramics. It is free from restraints and notches or other stress risers which should permit it to survive numerous thermal cycles associated with this application.

Extrusion appears to be an ideal forming method and should yield an ideal microstructure/property product. The work described under those two tasks has identified and demonstrated several useful aspects of this approach. However, it is recommended that further work be performed using the appropriate hot extrusion process which is better suited for PVB type binders. Limited small-scale trials have been made using a hot extrusion press at the GE Louisville Plastics Lab.

Although our success to date was limited, it is important to point out that a technology has been developed which has the potential for producing silicon nitride hardware which is felt to be vital for the successful development of a solar receiver and other applications. It is further to be noted that this abbreviated attempt to show feasibility was predicated on following essentially a high probability of success approach without room for alternatives, modifications, or restarts. With this goal in mind it would be simple to assume that the fabrication of such hardware is not possible. However, the lack of producing a final piece of hardware is the only negative result of this study. The lack of complete success was the direct result of a lack of flexibility and, most of all, time to evaluate and readjust approaches.

SECTION IV

REFERENCES

1. Robertson, C.S., et al., "A Conceptual Design Study of a High Temperature Solar Thermal Receiver", Final Report, JPL Contract 955455, General Electric Company, GEAD-66, January 4, 1980.
2. Nielsen, T.H., "Fabrication of Rectangular Cell Ceramic Regenerator for Use in Gas Turbine Engines", ASME Publication 77-GT-111, 1977.